



**PATENT APPLICATION**

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re the Application of

Toru Noguchi et al.

Group Art Unit: 1771

Application No.: 10/821,175

Examiner: COLE, ELIZABETH M

Filed: September 04, 2004

Docket No.: 127794

For: CARBON FIBER COMPOSITE MATERIAL AND  
PROCESS FOR PRODUCING THE SAME

**DECLARATION UNDER 37 C.F.R. §1.132**

I, Toru NOGUCHI, a citizen of Japan, hereby declare and state:

1. I have completed a degree in Doctoral Course in Materials Engineering, which was conferred upon me by the Graduate School of Science and Technology, Kobe University in Hyogo, Japan, in 1986. I have a B.S. degree in Metal Engineering, which was conferred upon me by the Department of Engineering, Tohoku University in Miyagi, Japan, in 1977.

2. I have been employed by Nissin Kogyo Co., Ltd. since 2002, and I have a total of 5 years of work and research experience in elastomers and metal composite materials. I was employed by Mitsuboshi Belting Ltd., from 1986 to 2001, and I have a total of 15 years of work and research experience, mainly in elastomer technology.

3. I am a member of The Japan Society of Polymer Science, The Japan Institute of Metals and The Japan Society of Applied Physics.

4. I am a named inventor in the above-captioned patent application.

5. I have a professional relationship with the assignee of the above-identified patent application. In the course of that professional relationship, I received compensation directly from the assignee for my work relating to Research and Development. I am being compensated for my work in connection with this Declaration.

6. This application describes a carbon fiber composite material comprising an elastomer and a carbon nanofiber substantially uniformly dispersed in the elastomer, wherein the elastomer has an unsaturated bond or a group, having affinity to the carbon nanofiber, wherein the elastomer in the composite material is in its uncrosslinked form, and has a spin-spin relaxation time ( $T_{2n}$ ) of its network component of 100 to 3,000  $\mu$  sec as measured at 150°C by the Hahn-echo method using pulsed NMR technique.

In contrast, a carbon nanofiber sulfonated in accordance with EXAMPLE 1 in Fisher et al, U.S. Patent No.6,203,814 is mixed in natural rubber by a conventional mixing method to obtain a compound in an uncrosslinked form which has a first spin-spin relaxation time ( $T_{2n}$ ) of 5,500  $\mu$ sec and a fraction ( $f_{nn}$ ) of components having the second spin-spin relaxation time of 0.45 as measured at 150°C by the Hahn-echo method using pulsed NMR technique. And a number of large sulfonated carbon nanofiber aggregates in the compound. Therefore, it was found that the sulfonated carbon nanofibers are not uniformly dispersed in the compound. Sulfonation of the carbon nanofiber and mixing of the carbon nanofiber and natural rubber were performed as follows.

First, the experiment on sulfonation of the carbon nanofiber will be described. A vapor grown carbon nanofiber having an average diameter of 87nm was used. The vapor phase reaction was carried out in a sealed glass flask. 20% fuming sulfuric acid was added to the vapor grown carbon nanofiber (60g) in the sealed glass flask. The vapor grown carbon nanofiber was stirred in 20% fuming sulfuric acid in the atmosphere in the range of temperature between 28°C and 30°C for 30 minutes and was subsequently discharged in ice water at pH 6.9 and filtrated to obtain a filter cake. The filter cake was dispersed and stirred in water and then filtrated. After repeating filtration three times, the filter cake was dried at 60°C for three days and the sulfonated carbon nanofibers (60g) was obtained. Fuming

sulfuric acid is viscous liquid and obtained by excess sulfur trioxide ( $\text{SO}_3$ ) absorbed by concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ). 20% indicated concentration of  $\text{SO}_3$ .

Second, the method of mixing the sulfonated carbon nanofiber and natural rubber will be described. Fisher does not teach a mixing method. Fisher recites "Without being bound to a particular theory, the functionalized fibrils are better dispersed into polymer systems" at column 7, lines 10 to 18. Thus, natural rubber was placed on the open roll with a roll distance of 2.0mm. The roll was not kept under temperature control. Subsequently, the sulfonated carbon nanofiber was mixed in natural rubber for 10 minutes and a compound was taken out. The roll temperature was about 90°C.

Moreover, the microscopic observation of tensile fractures of the compound revealed many aggregations of the sulfonated carbon nanofiber as shown Figs.1 to 5.

FIG.1

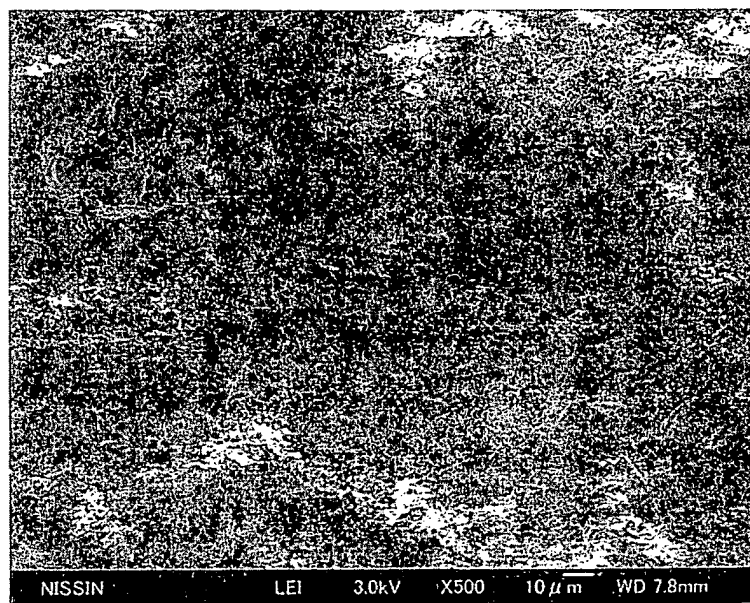


FIG.2



FIG.3

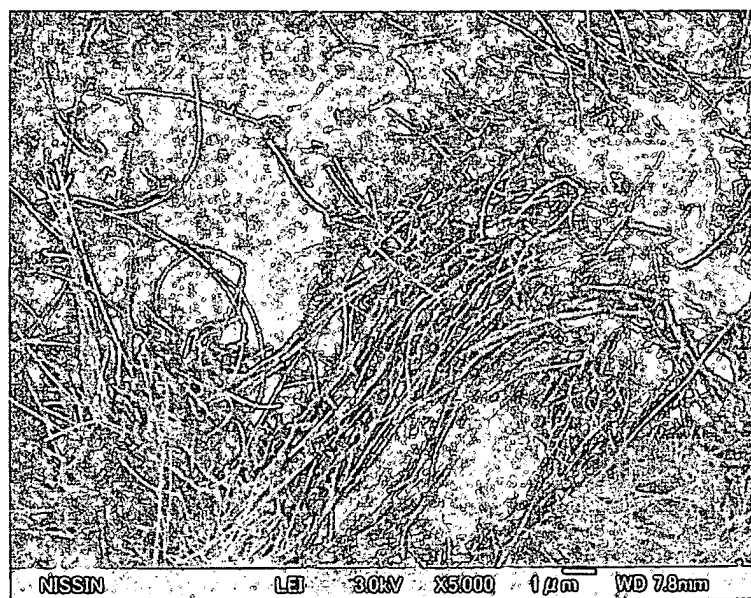


FIG.4

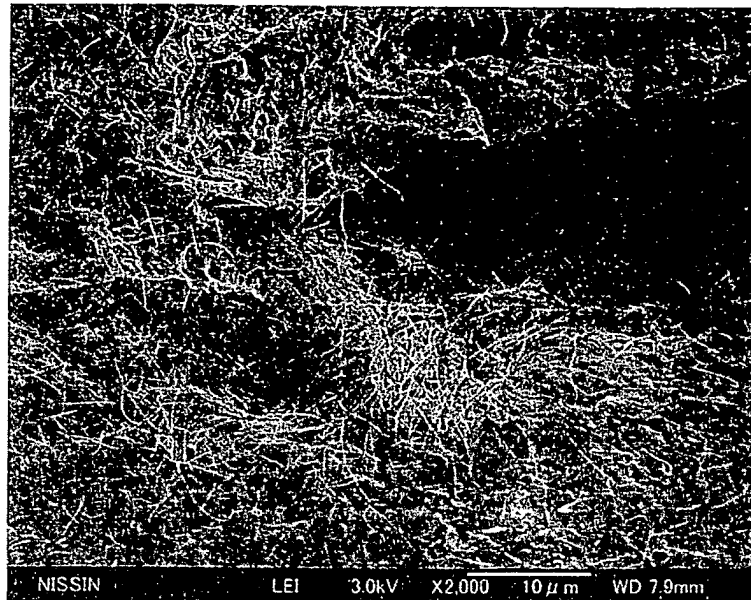
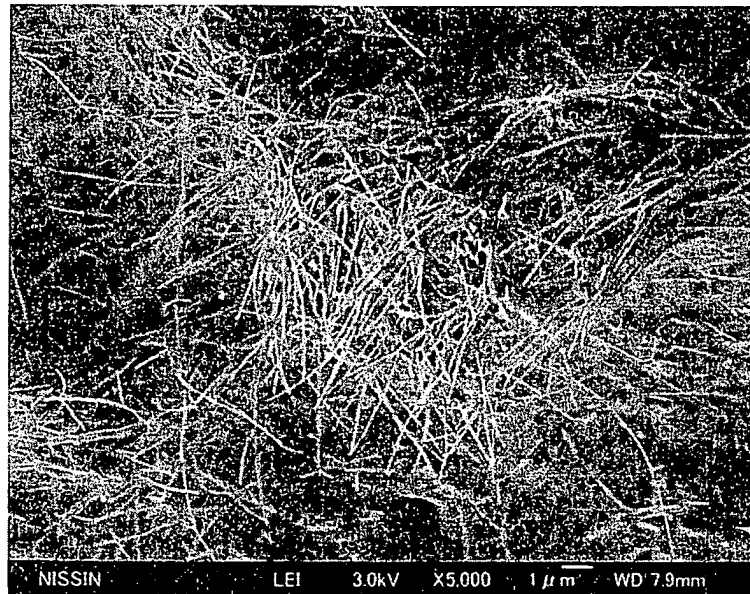


FIG.5



7. I hereby declare that all statements made herein are of my own knowledge and are true, and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and

the like so made are punishable by fine and/or imprisonment under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing therefrom.

Date: January 14, 2008

Toru Noguchi  
Toru NOGUCHI